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TECHNICAL REPORT

WHITE OAK LABORATORY

PHOTOLYSIS OF RDX IN AQUEOUS SOLUTION. INITIAL STUDIES

BY

Donald A. Kubose

John C. Hoffsommer

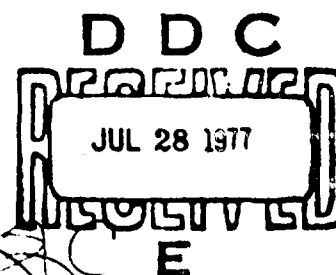
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23. ABSTRACT (Continue on reverse side if necessary and identify by block number) An experimental flow apparatus for the photolysis of RDX in aqueous solution is described. With this apparatus 98% of the RDX is destroyed at flow rates of 2500 ml/min. The end products formed include nitrogen gas, nitrous oxide gas, nitrate, and nitrite ions, formaldehyde and ammonia. One intermediate product has been identified as 1-nitroso-3,5-dinitro-1,3,5-triazacyclohexane. The primary photochemical steps involved in the photolysis are postulated. Areas for future work are outlined.												

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PHOTOLYSIS OF RDX IN AQUEOUS SOLUTION. INITIAL STUDIES

This report describes the use of photolysis to destroy RDX dissolved in waste water. The concepts and techniques developed during this study can provide the basis for a photochemical approach for decontamination of waste water containing photosensitive explosives and related substances. The work was performed under Task Number SF57-572-318/20392, Pollution Potential of Explosives in Water.

Julius W. Enig

JULIUS W. ENIG

By direction

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PHOTOLYSIS OF RDX IN AQUEOUS SOLUTION. INITIAL STUDIES

I. INTRODUCTION

In terms of pollution abatement the problems of treatment of RDX (1,3,5-trinitro-1,3,5-triazacyclohexane) contaminated waste water are different from those concerned with the treatment of TNT (2,4,6-trinitrotoluene) contaminated waste water. For example, RDX is not biodegradable as is TNT¹ and it is not adsorbed as readily as TNT on charcoal or polymeric adsorbents.^{1'}

A unique approach being studied at this Center concerns the use of strongly basic ion exchange resins to adsorb and chemically destroy RDX in a single step.^{1''}

Another very promising approach being actively pursued concurrently at this Center is the use of photolysis to degrade RDX in aqueous solution. The purpose of this report is to describe an experimental flow apparatus developed to study the geometric parameters involved in the photolysis of RDX contaminated water, to present results obtained to date on work performed to identify the photolytic products, and to suggest some photo-chemical mechanisms involved in the degradation of RDX.

II. FLOW PHOTOLYSIS SYSTEM

Cursory experiments using the full spectral output from a medium pressure mercury lamp (220 nm - 1367 nm) indicated that RDX in aqueous solution was rapidly destroyed by photolytic action.

¹Army Natick Laboratory, Conference on Microbiological Deterioration of Military Materiel, Natick, MA, Jun 1972, Technical Report 72-78 PR.

²G. Soli, NWC TP 5525, Jun 1973, Naval Weapons Center, China Lake, CA.

³B. W. Stevens, R. P. McDonnell (Rohm and Haas Co., Philadelphia, PA), R. K. Andren, and J. M. Nystrom (Army Natick Development Center, Natick, MA), paper presented at the 10th Annual Purdue Industrial Waste Conference, May 1975.

⁴W. B. Layne and J. Tash, Silas Mason Co., Inc., Middletown, IA, Feb 1976. Private communication.

⁵J. C. Hoffsommer, D. A. Kubose, and D. J. Glover, paper presented at the 2nd Annual Conference on Environmental Effects of Explosives and Explosions, Naval Surface Weapons Center, White Oak Laboratory, Silver Spring, MD, Oct 1976.

⁶D. A. Kubose, J. C. Hoffsommer, and H. I. Cordova, paper presented at the 2nd Annual Conference on Environmental Effects of Explosives and Explosions, Naval Surface Weapons Center, White Oak Laboratory, Silver Spring, MD, Oct 1976.

It was also found that if the spectral input to the RDX solution was limited to wavelengths greater than 280 nm by use of a pyrex filter, the rate of RDX disappearance was greatly reduced. To take advantage of this photolytic method for pollution-abatement, treatment of RDX contaminated water, several flow device ideas were tried which ultimately resulted in the design and construction of a laminar flow tray apparatus.

An important geometric parameter which must be examined is the path length or depth of solution to be photolyzed. Since the molecular extinction coefficient of RDX in aqueous solution is on the order of 10^4 at 220 nm and monotonically decreases to about 10^3 at 280 nm, a saturated solution of RDX in water at room temperature (approximately 40 ppm) is initially relatively opaque in that wavelength region.⁷ The rapidity at which this opacity disappears (due to destruction of the RDX) is shown by the results of an experiment in which tap water containing 29.5 ppm of RDX was placed in four separate 1-cm path-length quartz cells and the cells photolyzed such that the light passed consecutively from the first to the fourth cell. Table 1 shows the amounts of RDX left in each cell at various photolysis times.

TABLE 1
PHOTOLYSIS OF RDX SOLUTIONS CONTAINED IN CONSECUTIVE CELLS

Photolysis time sec	ppm RDX remaining in cell number			
	1	2	3	4
30	3.8	17.1	22.3	23.7
60	0.25	10.3	24.7	24.9
120	0.005	0.8	9.8	20.2

The effects of attenuation and scattering of the light caused by the windows of the cells must be kept in mind during examination of the results presented in Table 1.⁸ Thus, the concentrations of RDX remaining in the cells furthest from the light source are higher than they should be due to the presence of the cell windows.

On the basis of these results, the tray apparatus was designed with a variable height drain arrangement so that the real effect of changing the depth of the RDX solution could easily be determined.

⁷R. H. Jones and G. D. Thorn, Can. J. Research, 27B, 828 (1949)

⁸The extent of attenuation and scattering of the light is appreciable. The values of % transmission at 240 nm for 1, 2, 3, and 4 cells (filled with tap water) vs air are nominally 68, 52, 37, and 24, respectively, as determined on a Cary Model 16 spectrophotometer.

Figure 1 shows a schematic diagram of the tray apparatus. A "reverse waterfall" concept was used to achieve laminar flow characteristics in the tray. The RDX solution entered at the bottom of the vertical section, flowed up into the flat tray section, moved across the tray and exited through the three variable height drain tubes. Visual observation of the flow pattern, using ink as a marker, showed that the flow was laminar over the major portion of the tray area.

The tray was fabricated from 0.7-mm thick galvanized steel sheet. All joints were sealed with soft solder. The nominal dimensions of the flat tray section are 30 cm long, 14 cm wide and 4 cm deep. The nominal dimensions of the vertical section are 20 cm high, 14 cm wide and 2.5 cm thick. A 1200 watt medium pressure mercury lamp (Ace Glass Company, Vineland, New Jersey, 08360, Cat. No. 6531-10) with an active length of 30 cm was centrally located over the long dimension of the tray section approximately 1 - 2 cm above the RDX solution. A notch was cut in each side to accommodate the lower lamp heights for runs at the shallower solution depths. A reflector designed for use with the lamp (Ace Glass Company, Cat. No. 6531-02) covered the entire tray to maximize the light impinging on the RDX solution. The 1.4-cm ID drain tubes, made of pyrex, were held in tight fitting Teflon collars which were fastened to the bottom of the tray. The depth of solution in the tray was easily varied by adjusting the height of the tubes relative to the bottom of the tray by sliding them in their Teflon collars.

The aqueous RDX solution, made with tap water, was pumped from a 5-gallon jug by a peristaltic pump (Masterflex pump, Cole Parmer Instrument Company, Chicago, Illinois 60648, Cat. No. 7565).

Table 2 presents the results of a number of runs made at various flow rates and solution depths. The highest flow rate reported, 2500 ml/min, represents the limits of the tray design and of the pumping system used. Solution depths of less than 0.5 cm could not be achieved.

TABLE 2
RDX REMOVAL BY PHOTOLYSIS IN THE LAMINAR FLOW TRAY

% RDX removed at various flow rates					
Solution depth, cm	flow rate, ml/min				Initial RDX conc., ppm
	1000	1500	2000	2500	
0.5	99.9	99.8	98.7	96.9	22
1.0	99.9	99.9	98.6	96.9	39
2.0	99.9	99.9	99.5	97.3	26
3.0	99.9	99.8	99.6	98.9	18

The results in Table 2 show that RDX in aqueous solution is rapidly destroyed by photolysis in a flow system. An appreciation of just how rapidly the RDX is removed can be gained by calculating a residence time (i.e., photolysis time) of an element of solution in the tray. The shortest such residence time, obtained by dividing the solution volume in the tray by the flow rate, is 5 seconds for a depth of 0.5 cm and a flow rate of 2500 ml/min.

The one important fact to note in Table 2 is that the percent of RDX removed appears to be independent of solution depth (based upon data at the high flow rates). This shows that the photolytic decomposition products do not interfere by strongly absorbing in the wavelength region responsible for the degradation of RDX. Thus, the effect of deeper solution depths reflect only the increase in the residence time of the solution in the tray. For example, the percent of RDX removed remains constant (at a given flow rate) going from a depth of 0.5 cm to 3.0 cm since the residence time has increased proportionately. There even appears to be an increase in the efficiency of RDX removal at the higher flow rates at the 2 and 3 cm solution depths compared to the shallower depths.

The linear relationship between depth of solution and residence time, and the independence of the extent of RDX removal on solution depth suggests another, perhaps more efficient, photolysis geometry. The arrangement would simply be a deep box with the photolysis lamp centrally located immediately above the open top. The RDX solution would flow laminarily up from the bottom of the box and overflow over the top edges. This deep box geometry will allow maximum photolysis time for a given flow rate. The solution depths required for efficient removal of RDX at maximum flow rates will have to be determined experimentally.

III. PRODUCT ANALYSIS

The work on the photolytic apparatus described above has been complemented by efforts to determine the photodecomposition products of RDX in aqueous solution and attempts to understand the basic photochemical mechanisms taking place.

A. Experimental

1. Analytical Methods

RDX and other benzene extractable species were determined by vapor phase chromatography (vpc) using a Hewlett-Packard Model 5750 chromatograph equipped with a ^{63}Ni electron capture detector. The 4 ft x 1/4 in glass column was packed with 3.52% Dexsil GC on 80 - 100 mesh Chromosorb W AW DMCS and run at 180°C with 200 ml/min of 95:5 argon - methane carrier gas. The general analytical procedures used have been reported elsewhere.⁹

⁹J. C. Hoffsommer, J. Chromatog. 51, 243 (1970)

Mass spectra were obtained on a Varian Mat III gas chromatograph/mass spectrometer (gc/ms). Analyses of dissolved gases were performed on the Varian instrument using an apparatus constructed according to Swinnerton, et al.¹⁰

Analyses for nitrate ion were performed by conversion to nitrobenzene followed by vpc analysis of the nitrobenzene.¹¹ Nitrite ion determinations were carried out using the Griess method.¹²

Formaldehyde was determined by the chromotropic acid method described by Cares.¹³ Corrections were made for the presence of nitrate and nitrite ions.

Ammonia was determined by the Orion ammonia gas electrode (Orion Research, Inc., 380 Putnam Avenue, Cambridge, MA 02139).

Measurements of pH were made on a Beckman Model G pH meter.

2. Materials, Apparatus and Procedures

The RDX used in these studies was recrystallized twice from acetone. Aqueous solutions of RDX were made up in either distilled water or tap water by stirring the RDX in the water and filtering the solutions through a glass fiber filter prior to use.

All chemicals used were reagent grade, with the exception of benzene which was pesticide grade, and were used without further purification.

The general experimental setup used to photolyze the RDX solutions consisted of placing a quartz flask containing the RDX solution next to a quartz cooling jacket containing a 450 watt medium pressure mercury lamp (Ace Glass Company, Cat. No. 6515-34). Photolysis times were controlled by raising and lowering a cylindrical shutter around the quartz cooling jacket. When pyrex filtered light (i.e., wavelengths greater than 280 nm) was desired the RDX solution was placed in a pyrex flask or a cylindrical pyrex tube was placed around the 450 watt lamp. The RDX solutions were either aerated or deaerated by bubbling air or nitrogen, respectively, into the solutions. This bubbling also served to stir the solutions during photolysis.

¹⁰J. W. Swinnerton, V. J. Linnenbom and C. H. Cheek, Anal. Chem. 34, 483 (1962)

¹¹D. J. Glover and J. C. Hoffsommer, J. Chromatog., 94, 334 (1974)

¹²P. Griess, Ber. Deut. Chem. Ges. 12, 426 (1879); given in F. D. Snell and C. T. Snell, Colorimetric Methods of Analysis, Vol. 2, Van Nostrand, New York, 3rd ed., 1949, pp. 802-804

¹³J. W. Cares, Amer. Ind. Hyg. Assoc. 29, 405 (1968)

The general procedures employed in conducting an experiment consisted of the following. The concentration of RDX remaining in the photolyzed solution was determined by vpc analysis of a benzene extract of an aliquot of the solution. If further analysis of the aliquot was desired, e.g., nitrate or nitrite ion content, the aliquot was extracted three more times with benzene to quantitatively remove any residual RDX (a 1:1 benzene extraction removes 89% of RDX from an aqueous solution). This removal is necessary since any residual RDX will be hydrolyzed under the strongly acidic or basic conditions required for some of the analyses and the hydrolysis products will complicate the interpretation of the analyses.

A cursory search for dissolved gases was conducted by photolysis of the RDX solution in a specially constructed gas-tight syringe whose barrel was made of quartz. After photolysis the contents of the syringe was injected into a sample chamber which was in series with the carrier gas line of the Varian gc/ms (reference 10). Any dissolved gases were thus stripped from solution and entered the gc/ms for detection. A 2 ft x 1/8 in stainless steel column filled with Molecular Sieve 5A was run at 200°C for separation of the gases. Adequate precautions were taken to exclude atmospheric gases. RDX solutions made up in distilled water were used in these experiments.

B. Results

1. Analysis of Benzene Extract

The vpc trace of the benzene extract of a photolyzed RDX solution shows other peaks in addition to that due to residual RDX. The number and relative heights of these additional peaks vary according to the extent of photolysis, the spectral input (i.e., with or without a pyrex filter) and the pH of the solution during photolysis. The compounds responsible for these peaks are all photo-unstable since they become undetectable when the RDX becomes undetectable (nominally less than 0.005 ppm).

Exposure of the RDX solution to the full spectral output of the photolysis lamp (wavelengths greater than 220 nm) gives rise to the largest number of these peaks. As many as seven peaks with retention times (R_t) ranging from 0.2 min to 2.5 min ($R_t = 3.5$ min for RDX) are observable. The relative intensities of these peaks vary with the extent of photolysis but for a given photolysis time they are independent of dissolved oxygen content and solution pH. In contrast, when the spectral input to the solution is limited to wavelengths greater than 280 nm by use of a pyrex filter, the peak at $R_t = 2.5$ min is the only predominant one (compared to the relative intensities obtained at $\lambda > 220$ nm) and is present only when the pH of the solution is acidic. This observation is independent of dissolved oxygen content.

The pH of RDX solutions made up in distilled water falls to values of 3.5 - 4.0 when they are photolyzed with either pyrex filtered or unfiltered light from the photolysis lamp. When the solutions are made up in tap water the pH remains at 6.5 - 7.0 during photolysis with either filtered or unfiltered light. This buffering action is presumably due to the ions (e.g., bicarbonate) present in tap water. RDX solutions made up in distilled water and buffered at pH = 7.0 with a phosphate buffer gave the same results as RDX solutions made up in tap water.

A mass spectrum of the compound giving the peak at $R_t = 2.5$ min, obtained by gc/ms techniques, matched that of 1-nitroso-3,5-dinitro-1,3,5-triazacyclohexane (the mono-nitroso analog of RDX). Mass spectra of the other peaks imply similar type compounds but positive identification has been hampered by the very small amounts present and the lack of model compounds.

The absolute amounts of these compounds produced in the flow photolysis system described earlier is estimated to be very low. For example, at a solution depth of 3.0 cm and a flow rate of 2500 ml/min the amount of the mono-nitroso compound (it gives the most intense peak of all the compounds observed) is estimated to be less than 0.1 ppm.¹⁴ This represents less than 0.56% of the initial RDX concentration.

2. Analysis of Aqueous Portion after Extraction with Benzene

Varying amounts of nitrate ion, nitrite ion, formaldehyde, and ammonia have been found as products of RDX photolysis. The values of the mole ratios of these species to the moles of RDX photo-decomposed by unfiltered light (wavelengths greater than 220 nm) and pyrex filtered light (wavelengths greater than 280 nm), both at pH 7, are shown in Table 3. Also included in Table 3 are the mole ratio values for N_2O and N_2 . These gases were detected in "as photolyzed" distilled water solutions of RDX (i.e., without prior extraction with benzene). Another gaseous product which had a R_t greater than that of N_2O and whose peak height was about half that of N_2O was observed. It has not been positively identified yet, but CO_2 is the most probable candidate.

¹⁴ This value is based upon a ^{63}Ni electron capture detector response of 1.38 relative to RDX. Unpublished results, J. C. Hoffsommer, this Center.

TABLE 3
MOLE RATIO OF PRODUCTS TO RDX PHOTOLYZED

Ratio λ	NO_3^-	NO_2^-	CH_2O	NH_3	N_2O	N_2
	RDX	RDX	RDX	RDX	RDX	RDX
>220 nm	trace	2.4	0.8	0.6	0.05-0.1	0.1-0.2
>280 nm	0.7	2.0	0.6	0.7	---	---

C. Discussion

Although further work is required for a more complete product analysis, the data acquired to date permit some speculation as to the primary photochemical steps taking place. The differences in the products resulting from photolysis using unfiltered and pyrex filtered light from the mercury lamp suggests two different, wavelength dependent mechanisms.

It is postulated that at wavelengths greater than 280 nm (pyrex filtered light) the primary photochemical step is the homolytic cleavage of the nitramine bond to give the azayl radical and NO_2 . The interaction of the NO_2 with water provides a source of the observed nitrate and nitrite ions. Under acidic conditions (photolysis in distilled water solutions causes decrease in pH) the nitrite ion produces NO which can add to the azayl radical to give the observed mono-nitroso analog of RDX. When photolysis is carried out in buffered solutions or tap water, where the pH remains at or near neutral, the mono-nitroso compound is not observed since NO cannot be formed from nitrite ion. A run made in distilled water solution with added nitrite ion (hundred-fold excess over RDX) produced a five-fold increase in the amount of the mono-nitroso compound. This increase would be expected according to the above hypothesis.

When the incident wavelengths extend down to 220 nm (no pyrex filter) it is postulated that the predominant primary photochemical step is the cleavage of the N - O bond of the nitro group. This will give rise to the mono-nitroso compound directly and hence its formation will be independent of pH. This is what is observed. Additionally, the mono-nitroso compound can undergo further N - O bond cleavage to produce the di-nitroso analog of RDX. It is attractive to speculate that the additional peaks observed in the vpc analysis of the benzene extract are due to

this and other similar compounds. The addition of nitrite ion (same concentration as in pyrex filtered case) prior to photolysis produced only a 50% increase in the amount of the mono-nitroso compound. A small increase is not unexpected since the RDX solution is being exposed to all wavelengths emitted by the photolysis lamp. The very small amounts of nitrate ion observed compared to that found for wavelengths greater than 280 nm lend further support to the above hypothesis.

The presence of formaldehyde, ammonia, and the gases as products show that the ring is being broken. The paths from the primary photochemical steps to final products is undoubtedly complex and is yet to be resolved. The formation of nitrous acid must occur at some point since the pH of unbuffered solutions decreases asymptotically to about 4.

IV. FUTURE WORK

The use of photolytic means to degrade RDX in aqueous solution appears to be very promising. Additional work, both in terms of determining the most efficient photolysis geometry for handling RDX waste water (i.e., deep box geometry) and completing the product analysis/material balance studies is required. It is also desirable to extend and improve upon the present knowledge of the mechanisms involved in the photolysis of RDX in aqueous solution. The latter becomes important in assessing the efficacy of the photolytic process when other substances are present in solution with the RDX. A prime example is mixtures of TNT and RDX. Another example is determining whether the photolytic process can be enhanced by addition of specific substances such as hydrogen peroxide or ozone.

Work is in progress in the above areas and the results will be presented in future reports.

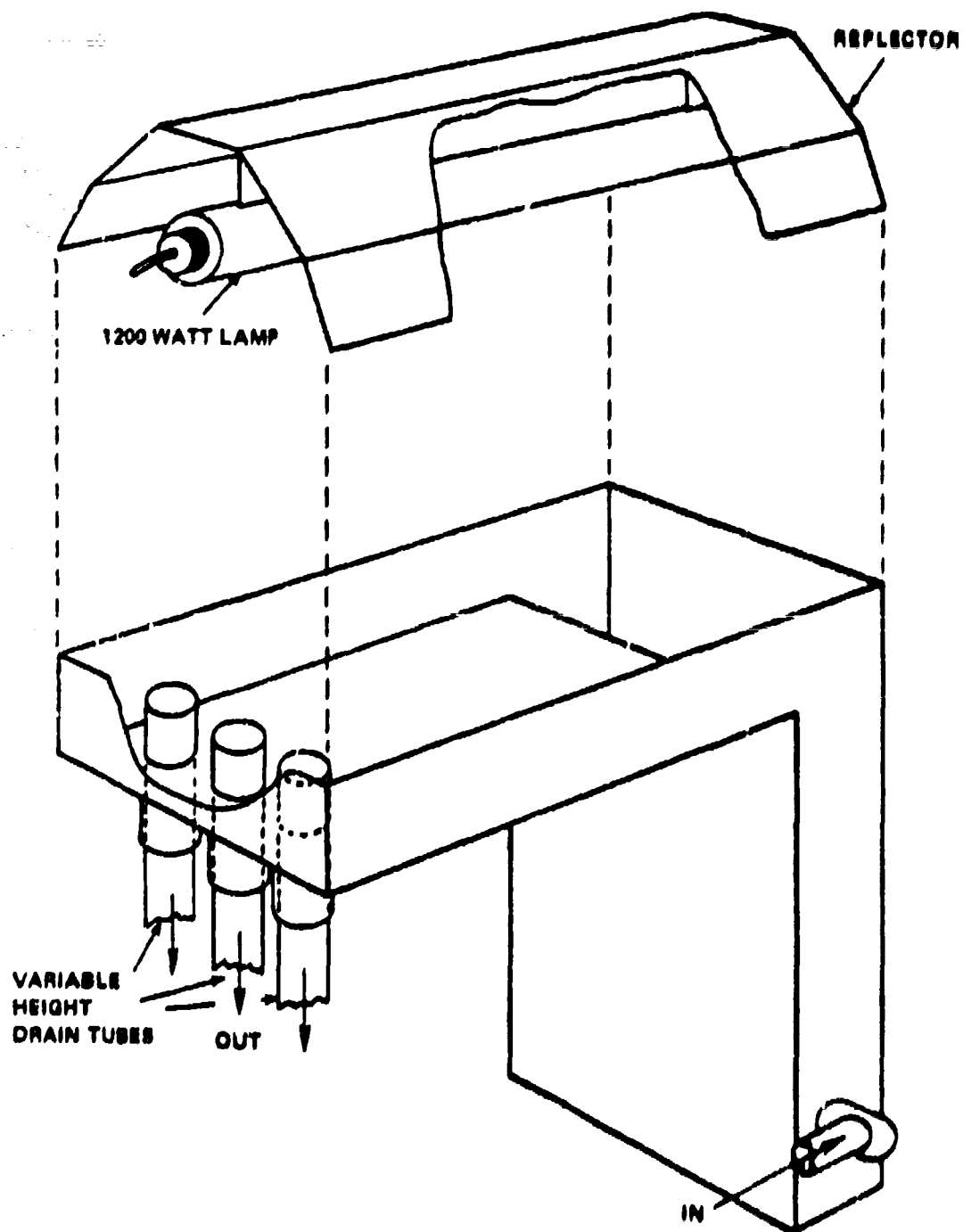


FIGURE 1
LAMINAR FLOW TRAY APPARATUS

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